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MEANS AND METHOD OF DEPOSITING THIN FILMS ON SUBSTRATES
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3,189,535

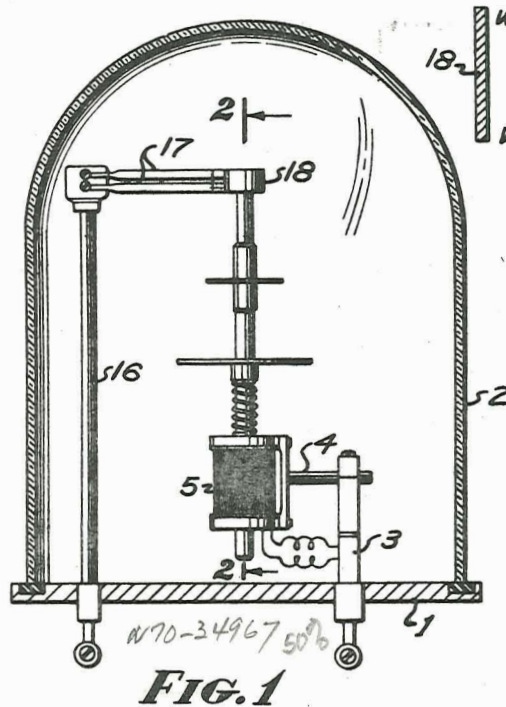


FIG. 1

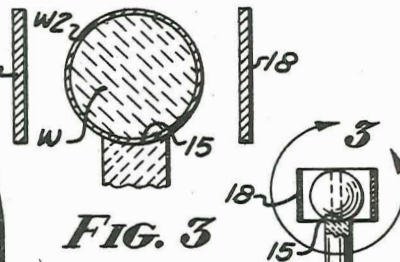


FIG. 3

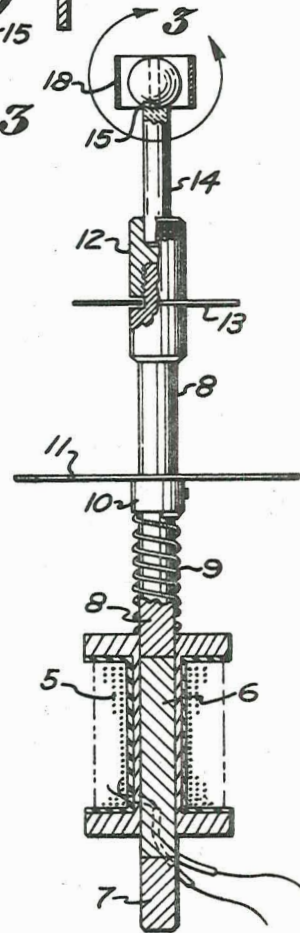


FIG. 2



FIG. 4

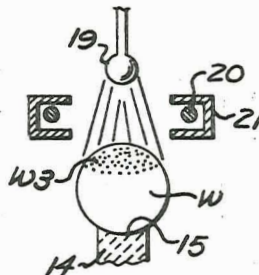


FIG. 5

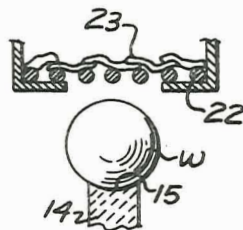


FIG. 6

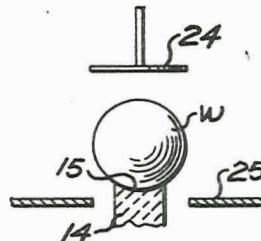


FIG. 7

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MEANS AND METHOD OF DEPOSITING THIN FILMS ON SUBSTRATES

James E. Webb, Administrator of the National Aeronautics and Space Administration, with respect to an invention of Hubert Erpenbach
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6 Claims. (Cl. 204—298)

This invention relates to means and method of depositing thin films on substrates, and included in the objects of this invention are:

First, to provide a means and method of depositing thin films on substrates which is particularly adapted to effect uniform deposition of thin metallic films of refractory metal on ceramic spheres at high temperatures.

Second, to provide a means and method of this class wherein the film itself may be applied by one of several techniques such as sublimation, electronic bombardment, thermal evaporation, or sputtering.

Third, to provide a means and method of this class wherein the ceramic sphere to be coated is vibrated and caused to rotate in a random manner so as to effect an even coating.

Fourth, to provide a means and method of coating ceramic spheres which are adapted to effect coating of the sphere with niobium.

With the above and other objects in view, as may appear hereinafter, reference is directed to the accompanying drawings in which:

FIGURE 1 is a diagrammatical view illustrating a bell jar, and showing one means of depositing thin films on substrates;

FIGURE 2 is an enlarged, partial sectional view, partial elevational view, taken through 2—2 of FIGURE 1;

FIGURE 3 is an enlarged, detailed sectional view taken within circle 3 of FIGURE 2, showing the workpiece, its support, and the surrounding means for effecting deposition of a metallic film on the workpiece;

FIGURE 4 is a view similar to FIGURE 3 indicating diagrammatically the zone of the workpiece which would be coated were it not for the vibration of the workpiece;

FIGURE 5 is a fragmentary, diagrammatical view similar to FIGURES 1 and 4, showing a modified means for effecting coating of the workpiece in which the coating is effected by electron bombardment;

FIGURE 6 is a similar fragmentary, diagrammatical view illustrating the arrangement to effect thermal evaporation of a metal to be deposited; and

FIGURE 7 is a similar diagrammatical view illustrating the arrangement utilized for sputtering the coated metal.

Reference is first directed to FIGURES 1, 2, 3 and 4. The apparatus here illustrated is in the nature of laboratory apparatus to simplify the illustration. The apparatus includes a base 1 on which is sealably fitted a bell jar 2, a mounting post 3 extends into the bell jar and carries a laterally extending bracket 4 which supports a solenoid coil 5.

The solenoid coil 5 receives an armature 6, the ends of which are attached to nonmagnetic shaft sections 7 and 8. The proportions of the solenoid coil and armature are such that when alternating current is applied to the solenoid coil, the armature 6 is caused to vibrate vertically. The mounting post 3 may also carry the conductors for supplying current to the solenoid coil 4.

A cooling coil 9 surrounds the shaft section 8 above the solenoid coil 5, this cooling coil is in the form of a tube. The ends of which extend outside the bell jar so

that a coolant may circulate therethrough from a source not shown.

The shaft section 8 is provided with a collar 10 above the cooling coil 9 which supports a lower heat shield 11.

The upper end of the shaft section 8 is reduced in diameter and externally screw threaded to receive an adapter sleeve 12. An upper heat shield 13 is loosely retained between the upper end of the shaft 8 and the adapter 12.

Secured within the upper end of the adapter sleeve 12 is a ceramic extension 14 which projects vertically thereabove. Its upper extremity is provided with a cavity 15 which preferably defines a spherical segment. The upper end of the ceramic extension 14 is adapted to support in stable equilibrium a spherical workpiece W.

A second mounting post 16 extends upwardly in the bell jar 2, that is provided with a laterally extending lead 17, the ends of which are connected to and support a split band 18 in the form of cylinder disposed at concentricity around the workpiece W. The band is formed of or is coated with the metal which is to be deposited on the workpiece W.

Operation of the apparatus shown in FIGURES 1 through 4 is as follows:

A spherical workpiece preferably formed of a non-conducting ceramic material is placed on the upper end of the ceramic extension 14. It is preferred that the workpiece have a smooth void free surface.

The material from which the workpiece is formed depends upon the use of which it is to be put. For example, it is desirable in the field of cryogenics to use a synthetic sapphire and coat the sapphire with a metal, such as niobium, usable under cryogenic conditions. The band 18 in this case is formed of niobium.

After the workpiece W and the band 18 have been properly positioned, the bell jar is set in place and a high vacuum is established within the bell jar. Current is then applied to the metal band sufficiently to raise its temperature to a point near melting point of the metal so that there is produced within the cylindrical space defined by the band, an atmosphere rich in the vapor of the metal from which the band is formed.

If the workpiece remained stationary within the metal vapor zone established by the band, a corresponding zone coating W1 of the workpiece would occur as shown in FIGURE 4. However, the solenoid coil is energized to cause vertical vibration of the armature and the ceramic extension 14 connected therewith. This causes the ceramic workpiece W to vibrate and to move randomly. The random movement of the workpiece is enhanced by the fact that the upper heat shield 13 is loosely supported which tends to produce lateral components of vibration. By reason of the random movement of the workpiece, all surfaces are coated and a remarkably uniform coating W2 is obtained.

Reference is now directed to FIGURE 5. Due to the fact that the workpiece is caused to vibrate and move randomly around its center, other means may be utilized to effect a coating. Thus as illustrated in FIGURE 5, the coating may be perfected by electronic bombardment. More specifically an anode 19 formed of the material to be evaporated on the workpiece is positioned above the workpiece. Between the anode 19 and the workpiece there is provided a focusing coil 20 surrounded by a shield 21. If the workpiece W did not vibrate the upper surface W3 only would be coated as indicated in FIGURE 5. However, by reason of the random vibration of the workpiece about its center, all portions are uniformly coated.

Reference is now directed to FIGURE 6. Instead of resistance heating of the material to be deposited on

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the workpiece, a grid heater 22 may be provided on which are placed small bits of the material to be evaporated on the workpiece as indicated by 23. Heat is applied to the grid heater to raise the material to be deposited to or near its melting point so as to create an atmosphere therebelow rich in the vapors of this material. Again due to the vibration of the workpiece all surfaces become coated.

Reference is directed to FIGURE 7. In this construction a sputtering technique is employed in which an anode 24 formed of the material to be deposited is placed above the workpiece and a cathode ring 25 is located below.

In utilizing the techniques indicated in the various figures, appropriate vacuum pressures are maintained within the bell jar and the atmosphere remaining is formed of an appropriate gas or gases.

While what hereinbefore has been described as the preferred embodiment of this invention, it is readily apparent that alterations and modifications may be resorted to without departing from the scope of this invention and such alterations and modifications are intended to be included within the scope of the appended claims.

What is claimed is:

1. Means for depositing thin metallic films on spheres, comprising:

- (a) a vertically disposed supporting rod recessed at its upper end to support a spherical workpiece with the major surface of the workpiece exposed above the supporting rod;
- (b) means for vibrating said rod thereby to vibrate said workpiece to cause random rotation thereof on said supporting rod;
- (c) and means for transforming a metal into a state capable of deposition and causing said metal to deposit on the surface of said randomly rotating workpiece.

2. Means for depositing thin metallic films on spheres as set forth in claim 1, wherein:

- (a) said workpiece and transforming means is disposed in a high vacuum enclosure;
- (b) and said transforming means includes a band of the metal to be deposited surrounding said spherical workpiece, means for heating said metal to evaporate the surface thereof, whereby an atmosphere of the vaporized metal is established around the workpiece.

3. Means for depositing thin metallic films on spheres as set forth in claim 1 wherein:

- (a) said workpiece and transforming means is disposed in evacuated enclosure;

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(b) and said transforming means includes a cathode formed of the material to be deposited, and a focusing means for directing electrons from said cathode to said workpiece.

4. Means for depositing thin metallic films on spheres as set forth in claim 1 wherein:

- (a) said workpiece and transforming means is disposed in evacuated enclosure;
- (b) and said transforming means includes a heater in proximity to said workpiece and in contact with said metal to effect thermal evaporation thereof.

5. Means for depositing thin metallic films on spheres as set forth in claim 1 wherein:

- (a) said workpiece and transforming means is disposed in evacuated enclosure;
- (b) and said transforming means includes a cathode formed of the metal to be deposited, and confronting the workpiece, and an anode surrounding said supporting rod.

6. Means for deposition of thin metallic films on spheres, comprising:

- (a) a vertically disposed supporting rod recessed at its upper end to support a spherical workpiece with the major surface of the workpiece exposed above the supporting rod;
- (b) means for vibrating said rod along a vertical axis;
- (c) a disk loosely retained by the upper portion of said rod to cause, as said rod is vibrated, lateral components of vibration whereby the upper end of said rod vibrates randomly, thereby to cause random rotation of said workpiece in its recess;
- (d) an enclosure for said workpiece rod and vibrating means, said enclosure adapted to be evacuated;
- (e) and means for transforming a metal into a state capable of deposition and causing said metal to deposit on the surface of said randomly rotating workpiece.

References Cited by the Examiner

UNITED STATES PATENTS

1,916,492	7/33	Schuricht	204—222
2,305,758	12/42	Berghaus et al.	204—192
2,822,301	2/58	Alexander et al.	117—107.1
2,960,457	11/60	Kuhlman	204—192
3,108,900	10/63	Papp	204—192

FOREIGN PATENTS

366,401 2/32 Great Britain.

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